

## PATENT ABSTRACTS OF JAPAN

(11)Publication number : 10-194710

(43)Date of publication of application : 28.07.1998

(51)Int.Cl.

C01B 15/10  
// C11D 3/395  
C11D 7/54

(21)Application number : 09-005535

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(22)Date of filing : 16.01.1997

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### (54) PRODUCTION OF SODIUM PERCARBONATE EXCELLENT IN SOLUBILITY

(57)Abstract:

PROBLEM TO BE SOLVED: To produce a sodium percarbonate composition, excellent in solubility in water and having expandability at the time of dissolution by coating grains of sodium percarbonate heated at a specific temperature with an alkali metallic (bi)carbonate and/or an alkali metal sulfate.

SOLUTION: A wet powdery sodium percarbonate with 6-15% moisture content and a binder are aggregated and granulated with a kneading type granulator. The grain size is then regulated with an extruder and the regulated grains are dried to afford sodium percarbonate grains having 300-2,000 $\mu$ m grain diameter. The resultant sodium percarbonate grains are subsequently arranged in a perforate plate and heated air is fed from the lower side to fluidize the sodium percarbonate grains and heat the grains at 95-105°C. A solution containing at least one metallic salt selected from an alkali metallic carbonate, an alkali metallic bicarbonate and an alkali metallic sulfate is then sprayed thereon to coat the surfaces of the sodium percarbonate grains with 3-40wt.% metallic salt described above.

### LEGAL STATUS

[Date of request for examination]

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

[Date of registration]

[Number of appeal against examiner's decision of rejection]

[Date of requesting appeal against examiner's decision of rejection]

[Date of extinction of right]

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**DETAILED DESCRIPTION**

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[Detailed Description of the Invention]

[0001]

[The technical field to which invention belongs] this invention is excellent in the solubility over water, and relates to the fault sodium carbonate which has foaming nature at the time of the dissolution. The fault sodium carbonate of this invention is blended with the detergent which mainly contains a bleaching agent constituent or a bleaching component, or is used suitable for home use or business use as an oxygen-evolution agent component.

[0002]

[Description of the Prior Art] A fault sodium carbonate is  $2\text{Na}_2\text{CO}_3$  and  $3\text{H}_2\text{O}_2$ . It is the sodium carbonate hydrogen-peroxide addition product expressed, it is mainly broadly used as an oxygen system bleaching agent component, and compared with a chlorine-based bleaching agent, since [ anxious about environmental pollution ] there is nothing, the amount used is being extended rapidly in recent years. Generally, in order to acquire the bleaching force as a chlorine system in which an oxygen system bleaching agent is the same since the bleaching force is low compared with a chlorine system, bleaching agent concentration higher \*\*\*\*\* at the time of use is needed. From a \*\*\*\* sodium carbonate being a powdered solid-state, the solubility which excelled at the time of dissolving is searched for.

[0003] On the other hand, although a fault sodium carbonate is not a combustible but a safe compound, when it contacts or mixes with a combustible, it may promote combustion under a certain conditions. Assistant inflammability needs to be decreased [ of this to a fault sodium carbonate ], and to add the diluent of mineral salt as the method is tried. For example, the method of making JP,58-204806,A containing a sodium chloride and a sodium sulfate in a fault sodium carbonate, and the method of blending a sodium bicarbonate with a fault sodium carbonate at JP,2-296705,A are indicated. However, if reduction of assistant inflammability of a fault sodium carbonate is performed by these methods, the solubility over the water of a fault sodium carbonate will get worse.

[0004] Moreover, when a fault sodium carbonate is sensitive and is blended with a detergent etc. to a water metallurgy group etc., the influence of the moisture in a detergent, the metal of the minute amount in the zeolite blended and a builder, etc. may decompose it quickly. Then, various the methods of covering a fault sodium-carbonate particle with mineral salt, and stabilizing it are also proposed. For example, in JP,57-7081,A, catalytic reaction of the front face of a fault sodium carbonate is carried out to alkaline-earth-metal salting-in liquid, the method of making the film of a carbonic acid alkali-metal salt forming in a front face, and the method of covering a fault sodium-carbonate particle front face in combination with a silicate and the carbonate of magnesium sulfate alkali metal to JP,7-17703,A are indicated, and it is covering of these mineral salt. Solubility got worse, and the stable fault sodium carbonate had the problem that sufficient effect could not be demonstrated, when it was said that it dissolves immediately and bleaching etc. is performed. .

[0005] The bleaching agent constituent containing the fault sodium-carbonate powder which some proposals also as the soluble improvement method are made, for example, was heat-treated 75 degrees C - 135 degrees C to JP,50-70286,A is indicated, it foams to the bleaching agent, and it is indicated that solubility is excellent. However, this aims at improvement in solubility and foaming nature about a fault sodium carbonate, it is not related with the stable fault sodium carbonate which covers mineral salt, and heat-treatment shows rather the inclination which promotes the assistant inflammability of a fault sodium carbonate.

[0006]

[Problem(s) to be Solved by the Invention] this invention is \*\* which is going to offer the fault sodium carbonate which was excellent in solubility and reduced assistant inflammability.

[0007]

[Means for Solving the Problem] This invention persons are making into 95 degrees C - 105 degrees C processing temperature at the time of covering a fault sodium-carbonate particle with at least one or more kinds chosen from the carbonic acid alkali-metal salt, the heavy carbonic acid alkali-metal salt, and the sulfuric-acid alkali-metal salt, and covering it, as a result of inquiring wholeheartedly, in order to solve this problem, found out that the fault sodium carbonate by which assistant inflammability was reduced was obtained, and completed this invention while they showed the outstanding solubility. .

[0008] It can be presumed that the solubility which was excellent while foaming when a part of available oxygen in a fault sodium carbonate decomposed at the time of covering, a cavity was made by keeping the temperature of a fault sodium carbonate high into a fault sodium-carbonate particle and this dissolved in water in case a fault sodium carbonate was covered with a metal salt is shown.

[0009] furthermore, the front face of the effect by the combustible and fault sodium carbonate other than reduction of assistant

inflammability according [ covering a fault sodium carbonate with a metal salt ] to the fall of available-oxygen concentration not contacting directly by covering, and a fault sodium carbonate -- a metal salt -- a wrap -- it is generated by decomposition of the fault sodium carbonate by things -- it is thought that they are synergisms, such as an effect which suppresses emission of oxygen gas

[0010]

[Embodiments of the Invention] Subsequently, with an extruder, condensation granulation of the fault sodium carbonate (6 - 15% of water contents) of the shape of a wet powder reacted, crystallized, dehydrated and manufactured by the well-known method is carried out with the granulating machine of a kneading formula with a binder, the particle size regulation of the fault sodium carbonate used by this invention is carried out, it is made to dry and it is obtained. Moreover, as for the fault sodium-carbonate particle before being covered, 300-2000 micrometers of 500-1000-micrometer things are usually preferably used for a path.

[0011] As the carbonic acid alkali-metal salt to cover, a heavy carbonic acid alkali-metal salt, and a sulfuric-acid alkali-metal salt, sodium salt and potassium salt can be illustrated and sodium salt is desirable from economical efficiency. The amount of covering of the alkali-metal salt to a fault sodium carbonate is 3 - 40 % of the weight, and is 10 - 25 % of the weight still more preferably five to 30% of the weight preferably. It is not desirable in respect of economical efficiency in \*\* to which the effect of assistant inflammability reduction will not be acquired if there are few amounts of covering than the range of appointed, but available-oxygen concentration will fall to if the amount of covering exceeds the range of appointed, and bleachability ability falls. The method of adding diluents, such as a carbonic acid alkali-metal salt, a heavy carbonic acid alkali-metal salt, and a sulfuric-acid alkali-metal salt, beforehand to the fault sodium-carbonate particle to cover, and covering with a metal salt further is also effective.

[0012] The method of covering coating sends the air which placed the fault sodium-carbonate particle on the perforated plate, and carried out lower shell heat, makes a fault sodium-carbonate particle flow, and although the method of spraying the solution of a metal salt is common, it is not limited to the above-mentioned method especially about the covering method. Processing temperature of the fault sodium carbonate at the time of covering is preferably performed at 100 degrees C - 105 degrees C 95 degrees C - 105 degrees C in order to produce and put a cavity into a fault sodium-carbonate particle. If the temperature of the fault sodium carbonate at the time of covering is less than the range of appointed, a cavity will not be made into a fault sodium-carbonate particle, and desired solubility will not be acquired. On the other hand, if temperature exceeds the range of appointed, decomposition of the available oxygen of a fault sodium carbonate will be large, and a fall will take place to an available oxygen quickly.

[0013]

[Example] An example and the example of comparison explain this invention in more detail. In addition, this invention is not limited to an example. Especially % display in an example and the example of comparison expresses weight %, as long as it is unstated.

[0014] The example 1 sodium carbonate and the hydrogen peroxide were made to react in solution, and the centrifugal separation machine separated the crystal which generated, it dehydrated, and the fault sodium carbonate of a damp or wet condition was obtained. The binder was added to the fault sodium carbonate of a damp or wet condition, water was added further, the water content was adjusted to 10%, it extruded with the granulating machine which attached the screen with a bore diameter of 1.0mm, and the granulation was performed, it dried and the fault sodium-carbonate particle of 600micro of mean particle diameters was obtained. (14.4% of available-oxygen concentration) It covered using fluidized-drying formula covering equipment (PAL screw coating equipment by Yamato science incorporated company) in 300g of obtained fault sodium-carbonate particles. From the attached spraying nozzle, the covering method puts in heating air from many \*\*\*\*, made the fault sodium-carbonate particle flow, it covered by having sprayed 270g of 20% solution of sodium sulfates, and maintained covering temperature for 20 minutes after that, and back-cooled. Processing temperature of the fault sodium carbonate at the time of covering was performed at 100 degrees C. The aggregate was not observed at all by the taken-out covering fault sodium-carbonate particle.

[0015] When it asks for the available-oxygen concentration of the obtained fault sodium-carbonate particle by the titration of a sodium thiosulfate, it is 11.6%, and the theoretical available-oxygen concentration of a covering fault sodium carbonate shows that a part of available oxygen of a fault sodium carbonate decomposed this available-oxygen value at the time of covering from  $14.4\% \times (1/(1+0.18)) = 12.1\%$  and the bird clapper. The dissolution examination and the combustion test were carried out using the obtained fault sodium carbonate. A test result is shown in Table 1. Dissolution time was 2 minutes, and it was dissolved, foaming. In a combustion test, combustion was not altogether carried out among the ten number of times of an examination.

[0016] 900ml of 20 degrees C (\*\*0.5 degrees C) water is put into a <soluble examination> 1l. beaker, a flag type agitator is stirred by 200rpm, 100g of samples is paid into it, change of the electrical conductivity of solution is read, and it considers as the time when the sample dissolved completely the time when electrical conductivity showed the highest value.

[0017] The sieve of 1180 micrometers of <combustion test> openings The passed sample is saved at the temperature of 20\*\*5 degrees C in the desiccator containing dryness silica gel for 24 hours or more. 24g of the sample, and the wood flour of the sapwood of the Japanese Japan cedar of 250 micrometer sieve passage of openings, and 500micro sieve interruption fault at 105 degrees C After 4-hour dryness, 6g of wood flour saved with the desiccator containing dryness silica gel for 24 hours or more is mixed, and the ratio of height and the diameter of a base puts into the cone cup of 1:1.75, turns this down on a heat insulation plate, makes conic deposition, and leaves it for 1 hour in the atmosphere of the temperature of 20\*\*5 degrees C, and 50\*\*10% of humidity. The circle cyclic nichrome wire heated at 1000 degrees C after that in the base of the deposition is contacted, and ignition and a combustion state are observed. Wood flour mixture is lit and the case where flaming continues to the last is considered as combustion. An examination is performed 10 times per one sample.

[0018] Using the fault sodium-carbonate particle used in the example 2 example 1, processing temperature was made into 105 degrees C, and also it covered like the example 1. The soluble examination of the obtained fault sodium-carbonate particle and the combustion test were carried out. Available-oxygen concentration is 9.4% and it foamed to it violently by the dissolution examination. A test result is shown in Table 1.

[0019] Using the fault sodium-carbonate particle used in the example 3 example 1, processing temperature was made into 95 degrees C, and also it covered like the example 1. The soluble examination of the obtained fault sodium-carbonate particle and the combustion test were carried out. Available-oxygen concentration is 11.9% and it foamed to it by the dissolution examination. A test result is shown in Table 1.

[0020] The sodium sulfate was changed into other compounds as coating using the fault sodium-carbonate particle used in the four to example 5 example 1, and also it covered like the example 1. The soluble examination of the obtained fault sodium-carbonate particle and the combustion test were carried out. The kind, the amount of covering, and test result of coating are shown in Table 1.

[0021] The sodium sulfate was covered 15% to the fault sodium carbonate using the fault sodium-carbonate particle which added the sodium bicarbonate 3% and it was corned to the fault sodium carbonate when corned the fault sodium carbonate of example 6 damp or wet condition, and also corned like the example 1 as covering material, and also it covered like the example 1. The dissolution examination of the obtained fault sodium-carbonate particle and a combustion test result are shown in Table 1.

[0022] The fault sodium-carbonate particle used in the example of comparison 1 example 1 was not covered, but the soluble examination and the combustion test were carried out. A test result is shown in Table 2. Although dissolution time was quick, in the combustion test, it burned altogether among 10 times.

[0023] Although the fault sodium carbonate was corned like example of comparison 2 example 1, 20% of sodium bicarbonate was added to the fault sodium carbonate at the time of a granulation. The soluble examination of the obtained fault sodium-carbonate particle and the combustion test were carried out. A test result is shown in Table 2. The dissolution rate was slow although combustion was not carried out.

[0024] The fault sodium carbonate of the example 2 of example of comparison 3 comparison was made to flow by hot blast, and heat-treatment was performed at 100 degrees C for 1 hour. Although dissolution time became early, it burned 6 times among 10 times in the combustion test, and assistant inflammability was promoted.

[0025] 20% of sodium-carbonate crystal (granular ash) was mixed with the V type blender to the fault sodium-carbonate particle and fault sodium carbonate which were used in the example of comparison 4 example 1. The soluble examination of the obtained fault-sodium-carbonate combination article and the combustion test were carried out. A test result is shown in Table 2. Dissolution time was late and burned 8 times among 10 times in the combustion test.

[0026] Using the fault sodium-carbonate particle used in the example of comparison 5 example 1, processing temperature was made into 85 degrees C, and also it covered like the example 1. The soluble examination of the obtained fault sodium-carbonate particle and the combustion test were carried out. A result is shown in Table 2. Although combustion was not carried out, the dissolution rate was slow and the foaming phenomenon was not seen.

[0027] Using the fault sodium-carbonate particle used in the example of comparison 6 example 1, processing temperature was made into 90 degrees C, and also it covered like the example 1. The soluble examination of the obtained fault sodium-carbonate particle and the combustion test were carried out. A result is shown in Table 2. Although combustion was not carried out, the dissolution rate was slow and the foaming phenomenon was not seen.

[0028] Using the fault sodium-carbonate particle used in the example of comparison 7 example 1, processing temperature was made into 110 degrees C, and also it covered like the example 1. The soluble examination of the obtained fault sodium-carbonate particle and the combustion test were carried out. A result is shown in Table 2. Although combustion was not carried out but being early foamed to the dissolution rate violently, the fall of available-oxygen concentration was large.

[0029]

[Effect of the Invention] Since it excels in the solubility over water and assistant inflammability is reduced, the fault sodium-carbonate constituent of this invention can be used suitable for a bleaching agent, a detergent, etc.

[0030]

[Table 1]

表1 実施例

No	条件			試験結果		
	被覆剤	過炭酸ナトリウム配合剤	被覆温度	有効酸素濃度	溶解時間	燃焼回数/試験回数
1	硫酸ナトリウム18%	-	100℃	11.6%	2分	0/10
2	硫酸ナトリウム18%	-	105℃	9.4%	1.5分	0/10
3	硫酸ナトリウム18%	-	95℃	11.9%	2.5分	0/10
4	炭酸ナトリウム20%	-	100℃	11.4%	2分	0/10
5	重炭酸ナトリウム15%	-	100℃	12.0%	1.5分	0/10
6	硫酸ナトリウム15%	重炭酸ナトリウム3%	100℃	11.7%	1.5分	0/10

[0031]

[Table 2]

表2 比較例

No	条件			試験結果		
	被覆剤	過炭酸ナトリウム配合剤	被覆強度 加熱処理	有効成分濃度	溶解時間	燃焼回数/ 試験回数
1	—	—	—	14.4%	1.5分	10/10
2	—	重炭酸ナトリウム20%	—	12.0%	5.5分	0/10
3	—	重炭酸ナトリウム20%	加熱処理 100℃	11.0%	3分	6/10
4	—	炭酸ナトリウム20%	—	11.9%	6分	8/10
5	硫酸ナトリウム18%	—	85℃	12.0%	3.5分	0/10
6	硫酸ナトリウム18%	—	90℃	12.0%	3分	0/10
7	硫酸ナトリウム18%	—	110℃	4.2%	1.5分	0/10

[Translation done.]